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(19)



(54) FABRIC-SOFTENING COMPOSITIONS

(71) We, UNILEVER LIMITED, a company organised under the laws of Great Britain, of Unilever House, Blackfriars, London E.C.4, England, do hereby declare the invention for which we pray that a patent may be granted to us and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to tertiary amine compositions useful as fabric softeners.

In recent years compositions capable of improving the softness of laundered fabrics have been sold for domestic use. Usually such fabric-softening compositions have taken the form of an aqueous solution or dispersion of a compound which confers a softening effect on fabric, and these solutions or dispersions are added to the water in which the fabric articles are rinsed after washing. A complete laundering process consists of a wash cycle in which a fabric is washed with an aqueous solution of a detergent composition, followed by removal of the bulk of the wash liquor from the fabric, and a rinse cycle, in which the fabric is rinsed with water to remove residual detergent composition, followed by removal of the rinse liquor. The rinse cycle may be repeated before the laundered fabric is dried. The need to add the softening composition during the rinse cycle can be inconvenient, particularly with automatic washing machines which would otherwise perform the complete laundering without interruption.

Attempts have therefore been made to develop a wash-cycle composition, that is, a composition to be added in the wash cycle and which is capable of softening fabrics in the presence of detergent-active compounds so that the fabric can be cleaned and softened simultaneously. This provides a difficult prob-

lem, for the active softening ingredient has to be capable of deposition in opposition to the action of the detergent whose function is to remove soil from the fabric, and in an amount sufficient to soften the fabric, but not so great that the appearance of the fabric is impaired. Furthermore, the active softening ingredient should not depress the detergent activity of the detergent-active compound in the composition, and most compounds which are known to be fabric softeners are cationic and form insoluble addition products with conventional anionic detergent-active compounds. Proposals have been made to solve this problem by using as softening agents certain betaines which undergo a charge reversal when the pH changes from the alkaline conditions of a wash liquor where they are anionic to the less alkaline conditions of a rinse liquor in which they are cationic and their deposition on to fabric is promoted. Such a betaine is soluble in the wash liquor and the small proportion of that used which is in the wash liquor residue present in the first rinse cycle has to suffice to provide the softening.

It has been proposed in German Patent 722,281 to use as textile softeners water-insoluble primary, secondary and tertiary fatty amines in conjunction with detergents and inorganic salts, the only tertiary amine specifically mentioned being dimethyldodecylamine. British Patent 1,052,847 describes the use in the wash cycle of solid fabric softeners which are complexes of urea and primary, secondary and tertiary amines having at least one straight-chain organic radical containing 12 to 18 carbon atoms, but no specific tertiary amines are mentioned.

It has now been discovered that a small group of tertiary fatty amines possess special

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advantages as wash-cycle fabric softeners because, like the betaines referred to above and unlike dimethyldodecylamine and tertiary or other fatty amines in general, they are negatively charged under the relatively strong alkaline conditions present in a typical wash cycle and therefore do not complex with anionic detergent-active compounds, but are protonated and therefore cationic and fabric-substantive under the neutral or slightly alkaline conditions present in a rinse cycle. They can therefore be added to an alkaline detergent wash liquor without reducing its cleaning capacity, and as they are insoluble in the wash liquor, when this is discharged most of the amine is left behind entrapped in the fabric so that it is available for softening in the rinse: this residual amine becomes substantive to the fabric when the rinsing water is added, so that before the rinse liquor is discharged it is adsorbed on to the fabric.

The isoelectric point of such an amine lies within narrow limits, for the change in its electronic properties, or charge reversal, has to occur between the pH of the wash liquor and the pH of the first rinse liquor, which is still weakly alkaline due to residual wash liquor. The pH of a wash liquor containing anionic detergent-active compounds is usually within the range from 8.0 to 9.9, depending on the detergency builder employed. It has been found that where the isoelectric point of the amine is substantially below the pH of the wash liquor and at least 0.2 units above the pH of the rinse water the charge reversal occurs on addition of rinse water to the fabric after discharge of the wash liquor. The amines having the desired properties are tertiary amines having an isoelectric point of from 8.3 to 9.8 and the structure RR^1R^2N where R is an alkyl group having from 1 to 6 carbon atoms and R^1 and R^2 are primary linear alkyl or alkenyl groups having from 10 to 26 carbon atoms.

The isoelectric point of any given amine can be determined by measuring the electrophoretic mobility of an aqueous dispersion of the amine under standard conditions of concentration and temperature and at a series of pHs, with plotting of the mobilities against pH to determine the pH of zero mobility. The isoelectric points of a series of fatty amines are given in the following table, in which T and Co represent the mixed alkyl radical derived from hydrogenated tallow acid and coconut oil acid, respectively. From this table it can be seen that those tertiary amines which are members of the above special group have the required isoelectric points, whereas the other amines do not.

Amines of the special group are in general old compounds, and their use has been described for purposes other than as fabric-softening agents. They can be prepared by the processes described in Kirk-Othmer's Encyc-

lopaedia of Chemical Technology, Volume 2, under Fatty Amines, or in US Patent 3,471,562, which describes the preparation of dioctadecylmethylamine and its use as an intermediate for the preparation of quaternary ammonium compounds which are themselves cationic fabric softeners but do not possess the property of charge reversal. British Patent 1,286,054 and US Patent 3,696,056 describe the use of fatty amines in compositions for controlling foam generated by synthetic detergent compositions and make no mention of fabric softening; the only tertiary amine having the structure given above that is specifically mentioned is a substance designated methyl dihydrogenated tallow amine.

T_2NH	7.4
T_3N	8.2
T_2NCH_3	8.8
$T_2N(CH_2)_3CH_3$	9.0
CO_2NCH_3	9.1
$T_2NCH_2CH_3$	9.4
$TN(CH_3)_2$	10.0
TNH_2	10.9

The only compositions disclosed containing that amine are ineffective for the purpose of providing fabric-softening, and in order to obtain the practical benefit of the present invention it has been found essential to formulate the fabric-softening amines in such a way as to provide compositions which are different from those described in British Patent 1,286,054 and US Patent 3,696,056.

According to the present invention, a fabric-softening composition comprises a tertiary amine having an isoelectric point of from 8.3 to 9.8 and of the structure RR^1R^2N where R is an alkyl group having from 1 to 6 carbon atoms and R^1 and R^2 are primary linear alkyl or alkenyl groups having from 10 to 26 carbon atoms, and a diluent, in which the diluent is water or a water-soluble non-surface-active salt; and where the diluent is water the composition is liquid and comprises from 2 to 50% of the amine dispersed in from 98 to 50% of water, by weight of the total amount of the amine and water, and where the diluent is a water-soluble non-surface-active salt, the composition is solid and comprises from 6.3 to 90% of the amine and from 10 to 93.7% of the salt by weight of the composition.

The tertiary amine softeners are preferably those where R has from 1 to 4 carbon atoms, and especially those where R is a methyl group. R will normally be a primary alkyl group. Preferably R^1 and R^2 each have from 12 to 22, and especially from 16 to 18, carbon atoms. R^1 and R^2 are linear groups but similar groups with minor amounts of branching insufficient to alter the character of the compounds as softeners are obvious chemical equivalents. Mixtures of amines can be employed, for instance commercial mixtures of compounds in which the alkyl or alkenyl

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groups are derived from the fatty acids of natural fats, as when coconut and hydrogenated tallow acids are converted to their nitriles and these are subsequently hydrogenated and alkylated. In accordance with usage in the fatty amine art, such mixed amines are here referred to as "coco" and "hydrogenated tallow" amines. R¹ and R² can be similarly derived from soyabean oil fatty acid. Preferably R¹ and R² are groups derived from a common source so that in single compounds they are the same group, and in commercial mixtures of compounds they represent the same individual groups in the same proportion.

Examples of suitable amines for use as wash-cycle softeners are:

Methyldidecylamine
Methyldilaurylamine
Methyldimyristylamine
Methyldicetylamine
Methyldimargarylamine
Methyldistearylamine
Methyldiarachidylamine
Methyldidocosylamine
Methylditetacosylamine
Methyldicocoamine
Methyldi(hydrogenated tallow)amine
Ethyldilaurylamine
Ethyldicetylamine
Propyldistearylamine
Butyldistearylamine
Ethyldicocoamine
Ethyldi(hydrogenated tallow)amine
Butyldi(hydrogenated tallow)amine
Methyldicocoamine and methyldi(hydrogenated tallow)amine are particularly suitable.

The invention provides a process for preparing an aqueous amine dispersion of the invention, in which the amine is dispersed in water in the presence of an emulsifier, preferably at a temperature at which the amine is in the liquid form. Suitable emulsifiers are listed in Kirk-Othmers' Encyclopaedia of Chemical Technology, Volume 8, page 128-130. The resulting emulsion may on cooling become a suspension due to solidification of the amine. Most dispersions thus prepared have a short life because they are unstable, and if stored before use are liable to separate into an aqueous phase and a solid amine phase: the successful preparation of dispersions that are stable for many weeks has presented a problem, for it has been found that the use of the standard procedures of emulsion technology for choosing an emulsifier system having the optimum hydrophiliclipophilic balance (HLB) fails to produce stable emulsions. It has however been discovered that satisfactorily stable dispersions can be prepared by introducing an emulsifier with a hydrophobic group similar to the hydrophobic groups of the amine: this appears to function by enabling

the formation of amine particles having at their surface the anionic polar head group of the emulsifier carrying a negative charge, thus altering the effective isoelectric point of the amine, while the packing of the hydrophobic group of the emulsifier with the corresponding groups of the amine enables retention of this group within the particles.

Accordingly a liquid composition of the invention preferably contains as emulsifier an anionic surface-active agent having a hydrophobic group that is a primary linear alkyl or alkenyl group having an average chain length within 2½ carbon atoms of the average chain length of the groups R¹ and R² in the amine. Particularly suitable are emulsifiers having a hydrophobic group distribution that is substantially the same as that of the amine. The anionic head group of the emulsifier can be carboxylate, sulphate, sulphonate, phosphate or phosphonate. Suitable emulsifiers can be chosen from sodium laurate, myristate, palmitate and stearate, disodium hexadecenyl succinate (which has an effective hydrophobic chain length of 16 carbon atoms), sodium N-tallow acyl glutamate (where the "tallow acyl" is a mixture of the acyl groups of tallow fatty acids, and the emulsifier has an effective hydrophobic chain length of 17 carbon atoms), sodium C₁₇-olefin sulphonate (having an effective hydrophobic chain length of 17 carbon atoms), sodium tallow fatty acid sulphonate (having an effective hydrophobic chain length of 17 carbon atoms) and sodium C₁₈-n-alkyl phosphate (having an effective hydrophobic chain length of 18 carbon atoms). Other emulsifiers which can be used are sodium hexylbenzene sulphonate and sodium octadecylbenzene sulphonate. Instead of sodium salts the salt of any equivalent cation providing water-solubility, for instance lithium, potassium or ammonium, can be used.

Where the amine employed is methyldicocoamine, the emulsifier is preferably sodium laurate, and especially the sodium salt of coconut oil fatty acid. Where the amine is methyldi(hydrogenated tallow)amine the emulsifier is preferably sodium stearate and especially the sodium salt of hydrogenated tallow acid.

Preferably the liquid amine composition contains from 5 to 30%, and especially from 7 to 25%, of the amine by weight of the total amount of amine and water, as such concentrations confer practical advantages in terms of correspondence to existing habits of users of liquid fabric conditioners having regard to the need to dispense a reasonable bulk of liquid in standard amounts and to provide an effective amount of amine in the wash liquor. An effective amount of the emulsifier is chosen from

within the range of 0.002 to 10% by weight of the dispersion, and generally from 0.1 to 20% by weight of the disperse phase is employed.

- 5 Preferably the solid amine composition contains at least 6.5, and especially at least 7%, of the amine. The amount of the salt present will usually be less than 75% by weight of the composition.

- 10 As water-soluble non-surface-active salts there are preferably used detergency builder salts. By "detergency builder salt" in this specification is meant a non-surface-active salt that possesses the power of increasing the detergent activity of a detergent-active compound. Suitable detergency builder salts are inorganic builders such as sodium ortho-, pyro-, trimeta- and tripoly- phosphates, sodium carbonate and sodium silicate. Examples of organic builders are salts of organic acids such as sodium citrate, sodium oxydiacetate, sodium carboxymethyloxysuccinate, sodium nitrilotriacetate and sodium ethylenediamine tetraacetate.

- 25 Non-surface-active salts other than detergency builders which can be used in solid compositions of the invention are sodium perborate or percarbonate which provide a bleaching function, and sodium sulphate.

- 30 Preferably where the composition contains a detergency builder salt, it also contains an anionic detergent-active compound, especially a non-soap (or synthetic) anionic detergent-active compound. Such a composition is preferably one containing from 6.5 to 35% of amine, from 3 to 60% of anionic detergent-active compound, and from 10 to 40 75% of detergency builder salt, by weight of the composition.

- Where a composition of the invention contains an anionic detergent-active compound, it can be a water-soluble or water-dispersible alkali metal salt of an organic acid, especially a sodium or potassium salt, or a corresponding ammonium or substituted ammonium salt. Examples of suitable organic acids are alkylbenzene sulphonic acids whose alkyl groups contain from 8 to 50 20 carbon atoms, for instance linear C_{10} - C_{15} alkylbenzene sulphonic acids; alkyl and alkenyl sulphonic acids of from 8 to 22 carbon atoms, for instance those known as olefin sulphonates which can be derived by reaction of sulphur trioxide with linear and branched olefins, especially "cracked wax" or "Ziegler" alpha-olefins, or those derived by reaction of alkanes with sulphur dioxide and chlorine and subsequent hydrolysis, or 60 by reaction of olefins with bisulphites; alkyl sulphosuccinates derived by reacting maleic acid esters with bisulphites; alkyl sulphuric acids of from 8 to 22 carbon atoms obtained 65 by reaction of alcohols and sulphur trioxide;

alkylether sulphuric acids obtained by reaction of molar quantities of alcohols of from 6 to 18 carbon atoms with 1 to 15 mols of ethylene oxide or mixtures of ethylene oxide and propylene oxide, and subsequently reacting the condensation product with sulphur trioxide; natural or synthetic aliphatic carboxylic acids of from 10 to 22 carbon atoms, especially the soaps obtained by splitting of triglyceride oils; and N-acylated isethionic and sarcosinic acids, where the acyl groups are those derived from such aliphatic carboxylic acids. Other suitable anionic detergent-active compounds are described in "Surface Active Agents", Volume I by Schwarz and Perry and "Surface Active Agents and Detergents", Volume II by Schwartz, Perry and Berch.

Compositions of the invention can contain other fabric-washing detergent composition ingredients, for instance lather boosters, such as coconut monoethanolamide; lather controllers; chlorine-releasing bleaching agents, for instance trichloroisocyanuric acid and sodium and potassium dichloroisocyanurates; antiredeposition agents, such as sodium carboxymethylcellulose; and perfumes, colourants, fluorescers, corrosion inhibitors, germicides and enzymes.

The invention provides a process for preparing a solid composition of the invention in which the amine is mixed with the non-surface-active salt and anionic detergent-active compound where present and, if required, the mixture is formed into granules. Preferably the solid composition is prepared as a spray-dried powder. Such a powder can be obtained by preparing an aqueous slurry containing the ingredients of the composition and heating the slurry to 80°C, at which the amine will be liquid and can with adequate mixing be distributed uniformly throughout the slurry, which is then spray-dried to give a powder containing evenly-distributed amine.

For domestic use, a liquid composition of the invention can be added in small quantities sufficient to provide softening, for instance from a bottle or a sealed sachet, for example from 10 to 100 ml, to a fabric load in a washing machine, together with a suitable detergent composition, for instance a washing powder. Solid compositions of the invention without the necessary detergent for a wash are added in the same way, and solid compositions containing detergent-active compound for a wash are used in the same way as normal detergent compositions. In order to get good results the pH of the wash liquor should be at least 0.2 pH units above the isoelectric point of the amine employed. This can be achieved by selection of a suitable amine for a given wash liquor or by adjusting the alkali content of the wash liquor, if required. The pH

of the wash liquor is preferably below 9.8 in order to avoid too great a buffering effect to enable the necessary reduction of pH in the rinse liquor to be made without having to lower the pH of natural rinse water: however, a wash liquor of high pH can be diluted with water after washing and before wash liquor discharge if it is desired to reduce the contribution of the residual wash liquor to the alkalinity of the first rinse liquor. For practical purposes the wash liquor pH should be above 8.5, so that scope is given for use of an amine whose isoelectric point is at least 0.2 units above the pH of the first rinse liquor.

The invention includes a process for the softening of fabrics, in which an aqueous anionic detergent wash liquor is prepared incorporating a composition of the invention, the liquor having a pH above the isoelectric point of the amine, fabric is washed with the wash liquor, and the pH of the wash liquor in contact with the washed fabric is then reduced to below the isoelectric point of the amine to attach the amine to the surface of the fabric. Preferably the bulk of wash liquor present during the washing step is separated from the washed fabric and the pH reduction is effected by the addition of rinse water to the fabric containing the residual wash liquor.

The invention also includes a softened fabric having a surface-coating of from 0.0005 to 0.01, and preferably from 0.001 to 0.005, parts per part by weight of the fabric, of a tertiary amine of the special group defined above having an isoelectric point of 8.3 to 9.8.

The invention is illustrated by the following Examples, in which all temperatures are in °C.

EXAMPLE 1

To a solution in water (84.85g) of sodium laurate (0.15g) at 80° was added molten methyldicocoamine (15g) and the mixture stirred and subjected to ultrasonic dispersion until it was homogeneous.

Soiled laundry (2.7kg) and 16 pieces (20.3cm square) of clean cotton towelling were placed in a front-loading automatic drum washing machine, followed by water (20 litres) at 50°, the amine dispersion (75ml) and a detergent product (100g) having the following composition in parts by weight:

Sodium dodecylbenzene sulphonate	7.5
Condensate of tallow fatty acid amine with 11 molar equivalents of ethylene oxide	3.5
Hardened tallow sodium soap	1.0
Sodium tripolyphosphate	64.9
Alkaline sodium silicate	1.0
Sodium sulphate	9.5
Sodium carboxymethyl cellulose	1.0
Sodium chloride	1.4

Fluorescer	0.2
Water	10.0

The resulting load had a liquor:cloth ratio of 7:1 with the wash liquor at pH 9.3. The fabrics were washed with a standard programme in which the load was heated to 95° with drum agitation during 35 min and then drained, leaving about 20% of residual wash liquor, and rinsed four times with cold water (20 litres, pH 7.5, giving pH 8.7 in the first rinse liquor), with spin-drying after the third and fourth rinse. The cloth was then allowed to dry in the atmosphere. The dried cloth had a surface-coating of about 0.002 parts of amine per part by weight of the fabric.

A similar operation was also carried out without the amine dispersion. The cloth pieces were assembled in pairs, one washed with amine and one without, and directly compared by a test panel of 5 operators who allotted an order of softening preference to each pair. In 77 out of 80 pair comparisons the cloth treated with the amine dispersion was selected as softer.

EXAMPLE 2

An amine dispersion was prepared as in Example 1, but using as amine methyldi(hydrogenated tallow)amine and as emulsifier sodium stearate.

Soiled laundry (2.7kg) and 16 pieces (20.3cm square) of cotton towelling was placed in a top-loading paddle washing machine, followed by water (68 litres) at 50°, the amine dispersion (50ml) and a detergent product (100g) having the following composition in parts by weight:

Sodium dodecylbenzene sulphonate	6.0
Condensate of hardened tallow fatty alcohol with 18 molar equivalents of ethylene oxide	4.0
Hardened tallow sodium soap	8.0
Sodium tripolyphosphate	33.0
Alkaline sodium silicate	8.0
Sodium sulphate	3.1
Sodium carboxymethyl cellulose	1.0
Sodium perborate	25.0
Fluorescer and perfume	0.7
Water	11.2

The resulting load had a liquor : cloth ratio of 25:1, with the wash liquor at pH 9.7. The fabrics were washed with a standard programme in which the load was heated to 95° with paddle agitation during 40 min and was drained and excess water removed by spinning, leaving about 10% of residual wash liquor, and rinsed twice with cold water (60 litres, pH 7.5, giving pH 8.2 in the first rinse liquor), with spin-drying after the final rinse, and the cloth allowed to dry in the atmosphere. The dried cloth had a surface-coating of about 0.0015 parts of amine by weight of the fabric.

The procedure was repeated using 32 pieces of towelling and no amine dispersion:

16 were removed and dried and the machine set for one more rinse and spin cycle, and 50ml of a commercial rinse conditioner containing 6% by weight of a mixture of dimethyldicocoammonium chloride and dimethyldi(hydrogenated tallow) ammonium chloride as softener was added and the 16 remaining pieces dried.

The 16 pieces from each treatment were arranged in triplets and submitted to a softness comparison by a 5-membered test panel. Each triplet was allotted a score of 1 for the softest piece, 2 for the next softest and 3 for the hardest. The total softness scores were as follows.

Cloths treated with	
amine dispersion	130
commercial rinse conditioner	113
detergent only	237

This demonstrates that softening with the amine dispersion used as a wash cycle softener was not significantly poorer than that with the commercial quaternary ammonium salt composition when used as a rinse cycle softener, both showing marked softening.

EXAMPLE 3

An amine dispersion was prepared as in Example 1, but using as amine methyl-di-stearylamine (isoelectric point 8.7) and as emulsifier sodium stearate.

As detergent there was used a composition having the following ingredients in parts by weight:

Sodium dodecylbenzene sulphonate	14.0
Coconut ethanolamide	2.0
Sodium tripolyphosphate	33.0
Alkaline sodium silicate	6.0
Sodium sulphate	17.0
Sodium carboxymethylcellulose	0.5
Sodium perborate	20.0
Fluorescer and perfume	0.2
Water	7.3

In a Tergotometer were placed 4 pieces of clean cotton towelling (20.3cm square), water (800ml), amine dispersion (2ml) and detergent composition (3.2g), giving a liquor : cloth ratio of 20:1, with the wash liquor at pH 9.7. The cloth was washed for 15 min at 80°, the wash liquor discharged, leaving about 10% as residue, the cloth rinsed twice in cold water (800ml, 24°C, pH 7.5, giving pH 8.2 in the first rinse liquor), and dried. The dried cloth had a surface-coating of about 0.002 parts of amine by weight of the fabric.

The procedure was also carried out using no amine dispersion. The sets of cloths were tested for softness in pairs as in Example 1. Every member of the panel found that the cloth treated with amine dispersion was softer than that treated with detergent alone.

EXAMPLES 4 and 5

An amine dispersion (Example 4) was

prepared as in Example 2, except that the amine used was a methyldialkylamine of isoelectric point 9.3, each of whose alkyl groups was derived from a commercial mixture of C₂₀ and C₂₂ straight chain saturated fatty acids in which the C₂₀ acid predominated.

Similar dispersions were prepared for comparisons using as amine tri(hydrogenated tallow)amine and dimethyl(hydrogenated tallow)amine whose isoelectric points are 8.2 and 10.0 respectively. The amine dispersion of Example 2 was also used (Example 5).

The washing procedure of Example 3 was carried out with each emulsion (with wash liquid pH 9.7 and first rinse liquor pH 8.2). The cloths were divided into 16 sets of 4 pieces including one washed with each emulsion, and graded by a test panel of 5 persons for softness, with scores allotted as 1 for the softest and 4 for the hardest. The combined softness scores obtained were:

Cloths treated with dispersions of methyl-di(C ₂₀ -C ₂₂ alkyl)amine	90
(Example 4)	136
methyl-di(hydrogenated tallow)amine	
(Example 5)	167
tri(hydrogenated tallow)amine	301
dimethyl(hydrogenated tallow)amine	196

The scores with the amine dispersions of examples 4 and 5 demonstrate a softening effect in comparison with those with the other dispersions. The cloths washed with the last dispersion had greasy deposits due to a precipitate formed from the amine and the anionic detergent used.

EXAMPLE 6

The amine dispersion of Example 2 was used as a wash cycle softener as in Example 3, in comparison with a commercial wash cycle softener containing 14% by weight of a quaternary imidazoline methyl sulphate, with the commercial quaternary ammonium salt rinse conditioner of Example 2, and also with the detergent composition of Example 3 with no added softener. The procedure used was similar to that in Example 3 except that 1ml of the amine emulsion of Example 2 and 1ml of the quaternary imidazoline salt softener were used as wash cycle softeners, and washed were also carried out with the detergent composition with no softener both with and without the commercial rinse conditioner of Example 2 (1ml) added to the last rinse liquor. The resulting dried cloths were panel-tested as before, and the following softness scores obtained.

Cloths treated with dispersion of methyl-di(hydrogenated tallow)amine	108	130
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commercial wash cycle softener 270
commercial rinse conditioner 142
detergent only 280

5 EXAMPLES 7 to 15

Amine dispersions were prepared as in Example 2, but using a range of amine concentrations, namely 5, 10, 20, 25, 30, 35, 40, 45 and 50% of methyl-di(hydrogenated tallow)amine by weight of the dispersion and sodium stearate as emulsifier. The emulsions were found to be stable for at least 6 weeks at ambient temperature.

10 The process of Example 3 using an amine dispersion but with the amine dispersion replaced by 1ml of the 30% dispersion above (Example 11) was carried out and softened cotton fabric produced.

20 EXAMPLES 16 to 20

Amine dispersions were prepared as in Example 2, but using a range of emulsifier concentrations, namely 0.015, 0.45, 0.75, 1.5 and 3% of sodium stearate by weight of the dispersion. The dispersions were found to be stable for at least 6 weeks at ambient temperature, and that with 0.45% emulsifier was stable for at least 6 months.

30 EXAMPLES 21 to 30

Amine dispersions were prepared as in Example 2, but using a series of different emulsifiers, and the period of stability of each emulsion at 20° was observed, as follows.

Example	Emulsifier	Stability
21	sodium laurate	10 days
22	sodium myristate	2 weeks
23	sodium palmitate	at least 6 weeks
40	24 sodium hexadecenyl succinate	at least 10 weeks
25	sodium N-tallow acyl glutamate	4 weeks
45	26 sodium C ₁₆ -C ₁₈ α-olefin sulphonate	4 weeks
27	sodium hexylbenzene sulphonate	at least 10 weeks
28	sodium octadecylbenzene sulphonate	4 weeks
50	29 sodium tallow fatty acid sulphonate	at least 10 weeks
30	sodium C ₁₆ -C ₂₀ n-alkyl phosphate	6 weeks

55 EXAMPLES 31 and 32

Stable amine dispersions are prepared as in Example 1 but using as amine and emulsifier respectively methyl-dicocoamine and the sodium salt of coconut oil fatty acid: and methyl-di(hydrogenated tallow)amine and the sodium salt of hydrogenated tallow fatty acid.

60 EXAMPLE 33

65 A solid fabric-softening composition hav-

ing the following ingredients in parts by weight was prepared.

Sodium dodecylbenzene sulphonate	14.0	
Coconut ethanolamide	2.0	
Sodium tripolyphosphate	33.0	70
Sodium sulphate	6.9	
Alkaline sodium silicate	6.0	
Methyl-di(hydrogenated tallow)amine	10.0	
Sodium carboxymethylcellulose	0.5	75
Fluorescer	0.4	
Sodium perborate	20.0	
Water	7.2	

All the ingredients except the amine and the perborate were mixed together with an additional amount of water (62 parts) to provide a slurry, and heated to 80°, the amine was added in the molten state and the mixture thoroughly stirred and spray-dried to give a dry free-flowing powder, with which the perborate was finally mixed.

The composition was used to wash cotton pieces in a Tergotometer at 0.4% concentration as in Example 3, with a wash liquor pH of 9.7 and a first rinse liquor pH of 8.3. The dried cloth had a surface-coating of about 0.004 parts of amine by weight of the fabric. A control wash was also carried out using a similar composition in which the amine was replaced by the same amount of additional sodium sulphate. Comparison by a test panel as before was made and the cloth treated with amine was chosen as softer in every instance.

100 EXAMPLE 34

A solid fabric-softening composition was prepared having the same ingredients as the detergent product of Example 1 except that 7.5 parts of methyl-di(hydrogenated tallow)amine was substituted for the same amount of sodium sulphate. The composition was prepared by mixing together all the ingredients except the amine and a portion (23 parts) of the sodium tripolyphosphate, together with an additional amount of water (60 parts) to provide a slurry, heating the mixture to 80°, adding the molten amine, stirring the mixture thoroughly and spray-drying to give a dry free-flowing powder, with which the remaining tripolyphosphate was mixed.

The composition was used to wash cotton pieces in a Tergotometer as in Example 3 at 0.2, 0.4 and 0.6% concentrations, with a wash liquor pH of 9.3 and a first rinse liquor pH of 8.1, and control washes were also carried out using the detergent product of Example 1. Comparison by a test panel as before showed that the amine-treated cloth was softer than the control in every instance.

120 EXAMPLE 35

A composition is prepared as in Example 34, except that the amine employed is 130

methyldicocoamine: 100g of the composition is used in a front-loading automatic washing machine in a laundering process similar to that described in Example 1, with a wash liquor pH of 9.3 and a first rinse liquor pH of 8.7, to provide softening of the cotton fabric.

WHAT WE CLAIM IS:—

1. A fabric-softening composition comprising a tertiary amine having an isoelectric point of from 8.3 to 9.8 and of the structure $RR'R^2N$ where R is an alkyl group having from 1 to 6 carbon atoms and R^1 and R^2 are primary linear alkyl or alkenyl groups having from 10 to 26 carbon atoms, and a diluent, in which the diluent is water or a water-soluble non-surface-active salt; and where the diluent is water the composition is liquid and comprises from 2 to 50% of the amine dispersed in from 98 to 50% of water, by weight of the total amount of amine and water, and where the diluent is a water-soluble non-surface-active salt, the composition is solid and comprises from 6.3 to 90% of the amine and from 10 to 93.7% of the salt by weight of the composition.
2. A composition according to Claim 1, where R in the amine has from 1 to 4 carbon atoms.
3. A composition according to Claim 2, where R is a methyl group.
4. A composition according to any preceding claim, where R^1 and R^2 each have from 12 to 22 carbon atoms.
5. A composition according to Claim 4, where R^1 and R^2 each have from 16 to 18 carbon atoms.
6. A composition according to Claim 3, where the amine is methyldicocoamine.
7. A composition according to Claim 3, where the amine is methyldi(hydrogenated tallow)amine.
8. A composition according to any preceding claim, in which the diluent is water.
9. A composition according to Claim 8, and containing from 5 to 30% of the amine.
10. A composition according to Claim 9, and containing from 7 to 25% of the amine.
11. A composition according to any one of Claims 8 to 10, and containing as emulsifier an anionic surface-active agent having a hydrophobic group that is a primary linear alkyl or alkenyl group having an average chain length within $2\frac{1}{2}$ carbon atoms of the average chain length of the groups R^1 and R^2 in the amine.
12. A composition according to Claim 11, where the amine is methyldicocoamine and the emulsifier is sodium laurate.
13. A composition according to Claim 11, where the amine is methyldi(hydrogenated tallow)amine and the emulsifier is sodium stearate.
14. A composition according to Claim 11, in which the emulsifier has a hydrophobic group distribution that is substantially the same as that of the amine.
15. A composition according to Claim 14, where the amine is methyldicocoamine and the emulsifier is the sodium salt of coconut oil fatty acid.
16. A composition according to Claim 14, where the amine is methyldi(hydrogenated tallow)amine and the emulsifier is the sodium salt of hydrogenated tallow acid.
17. A composition according to any one of Claims 1 to 7, in which the diluent is a water-soluble non-surface-active salt.
18. A composition according to Claim 17, and containing at least 7% of the amine.
19. A composition according to Claim 17 or Claim 18, in which the salt is a detergency builder salt.
20. A composition according to Claim 19, and containing an anionic detergent-active compound.
21. A composition according to Claim 20, and containing from 6.5 to 35% of the amine, from 3 to 60% of anionic detergent-active compound, and from 10 to 75% of detergency builder salt, by weight of the composition.
22. A composition according to any one of Claims 17 to 21, and containing as salt sodium tripolyphosphate.
23. A composition according to any one of Claims 17 to 22, and containing as salt sodium perborate or percarbonate.
24. A composition according to any one of Claims 17 to 23, and containing as salt sodium sulphate.
25. A composition according to Claim 1, substantially as described in any one of the accompanying Examples.
26. A process of preparing a composition according to any preceding claim, in which the amine is dispersed in water in the presence of an emulsifier or is mixed with the water-soluble non-surface-active salt.
27. A process according to Claim 26, substantially as described in any one of the accompanying Examples.
28. A process for the softening of fabrics, in which an aqueous anionic detergent wash liquor is prepared incorporating a composition according to any one of Claims 1 to 25, the liquor having a pH above the isoelectric point of the amine, fabric is washed with the wash liquor, and the pH of the wash liquor in contact with the washed fabric is then reduced to below the isoelectric point of the amine to attach the amine to the surface of the fabric.
29. A process according to Claim 28, in which the bulk of wash liquor present during the washing step is separated from the washed fabric and the pH reduction is effected by the addition of rinse water to the

fabric containing the residual wash liquor.

30. A process according to Claim 29, substantially as described in any one of Examples 1 to 6, 11 and 33 to 35.

5 31. A softened fabric when obtained by a process according to any one of Claims 28 to 30.

32. A softened fabric having a surface-coating of from 0.0005 to 0.01 parts, per part by weight of the fabric, of an amine as defined in Claim 1. 10

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